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Synthesis of compounds

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An abbreviated version of this protocol was published in Science Advances in Jan 2020

Modified cyclodextrins as broad-spectrum antivirals

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Detailed protocol

All solvents used were dry, and reactions were carried out under argon atmosphere. The starting materials were purchased from Sigma-Aldrich if not otherwise stated. Heptakis-(6-deoxy-6-mercapto)-beta-CD and hexakis-(6-deoxy-6-mercapto)-beta-CD were purchased from Cyclodextrin-Shop, Netherlands. Care was taken to use a freshly synthesized batch to minimize the presence of disulfides.

Synthesis of sodium undec-10-ene-1-sulfonate
- 11-Bromo-1-undecene (2.122 g, 9.1 mmol) and Na_2SO_3 (3.061 g, 24.3 mmol) were added into a mixture of MeOH (25 ml) and H_2O (45 ml) and refluxed for 18 hours.
- MeOH was removed under reduced pressure vacuo, and the aqueous layer was washed with Et_2O (2 Â~ 50 ml) and dried.
- The white solid was extracted with hot MeOH and filtered twice, leaving the insoluble Na_2SO_3 salt on the filter.
- The filtrate was recrystallized from EtOH/H_2O mixture (20/1), and the product was collected as white crystalline needles (1.983 g, 85%).

Synthesis of CD1
- Heptakis-(6-deoxy-6-mercapto)-beta-CD (50 mg, 0.040 mmol), sodium undec-10-ene-1-sulfonate (108 mg, 0.421 mmol), and 2,2-dimethoxy-2-phenylacetophenone (22 mg, 0.084 mmol) were dissolved in DMSO (5 ml).
- The reaction mixture was placed under ultraviolet (UV) lamp (250 W) and stirred for 18 hours.
- Crude product was precipitated by the addition of Et_2O (45 ml) and collected by centrifugation.
- The off-white solid was washed by MeOH (45 ml) and EtOH (45 ml) and collected by centrifugation.
- The product was purified by dialysis against Milli-Q H_2O for 3 days, filtered through 0.2-□m filter, and collected as a yellow solid (92 mg, 76%).

How to cite:(Readers should cite both the Bio-protocol preprint and the original research article where this protocol was used)

1. Jones, S. T.(2020). Synthesis of compounds. Bio-protocol Preprint. bio-protocol.org/prep320.
2. Jones, S. T., Cagno, V., Janeček, M., Ortiz, D., Gasilova, N., Piret, J., Gasbarri, M., Constant, D. A., Han, Y., Vuković, L., Král, P., Kaiser, L., Huang, S., Constant, S., Kirkegaard, K., Boivin, G., Stellacci, F. and Tapparel, C.(2020). Modified cyclodextrins as broad-spectrum antivirals . Science Advances 6(5). DOI: 10.1126/sciadv.aax9318